OIPE
AUG 1 2 2005
JC110
PATENT & TRADEMARK OFFICE

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In Re Application of: Pickard et al.	Docket No.: 7000Z
Serial No.: 10/677,454	Group Art Unit: 1742
Filed: October 2, 2003	Examiner: Ngocian T. Mai

For: High Thermal Conductivity Metal Matrix Composites

DECLARATION UNDER 37 CFR 1.132 OF ROGER S. STORM

I, Roger S. Storm, hereby declare that:

1. I reside at 4421 N. Palisade Drive, Tucson, Arizona 85749.

2. I earned a B.A. in Chemistry in 1964 from SUNYAB, and a Ph.D. in Physical Chemistry in 1969 from SUNYAB.

3. I have had over 40 years of extensive experience in both hands on experimentation and management of advanced materials research and development, including polymers, ceramics, metals, and composites.

4. Since 2001, I have been employed by MER Corporation as Senior Vice President and technical manager of high thermal conductivity composites.

5. I am familiar with the subject matter of both the above-identified U.S. Patent Application and the Miyamoto et al. U.S. Patent No. 6,673,439.

6. In order to duplicate for thermal conductivity measurements the metal matrix composite materials containing SiC coated diamond particles as described in the Miyamoto et al. U.S. Patent No. 6,673,439, the following experiments were conducted under my supervision and direction.

7. A first sample of SiC coated diamond powder was prepared using the Miyamoto et al. process for conversion coating of diamond with SiO to duplicate Inventive Example 2 in Table 1 in Example 1 of the patent. An alumina crucible and lid was purchased. Grafoil was used for the carbon paper. A piece of grafoil was made to cover the bottom of the alumina crucible and another piece of grafoil was made to cover the circular walls of the crucible. A quantity of 4.76 grams of commercially available silicon monoxide (SiO) powder from Alfa-Aesar with a particle size of 400 microns or less was weighed out and placed on top of the grafoil in the bottom of the crucible. Next, a disk of 1/8" thick graphite felt was placed on top of the silicon monoxide powder. A quantity of 14.17 grams of SAT MS4-M diamond powder with a particle size of 30-40 microns was poured onto the 1/8" graphite felt. The ratio of diamond powder weight to silicon monoxide weight was 2.52, within the range

specified in Miyamoto et al., column 5, lines 50-52. Then another piece of grafoil was placed on top of the diamond powder, but not touching the diamond powder. The alumina lid was then placed on the crucible, such that it could be subsequently slipped off.
The loaded crucible was then placed in a Centorr vacuum furnace. The run conditions were a 2 hours ramp from room temperature to 1300°C, followed by a 2 hour soak at 1300°C. The sample was purged with Argon gas and run under vacuum of .01 torr or higher. This run was made on Wednesday, May 4, 2005. The top grafoil was removed from the crucible and the diamond powder carefully poured out. An XRD and SEM of the reacted powder were obtained.

8. A second sample of SiC coated diamond powder was produced using the chemical vapor deposition (CVD) coating process to duplicate Comparative Example 5 in Table 1 in Example 1 of the patent. A quantity of 3 grams of SAT MS4-M diamond (30-40 micron particle size) was placed in a graphite mesh basket. The reactor was induction heated using a graphite susceptor to 1300-1305°C at a pressure of 90 torr. A flow rate of 50 sccm (standard cubic centimeters per minute) of methyltrichlorosilane (MTS) with a flow rate of 150 sccm H_2 carrier gas was maintained for a period of 1 hour. A second run was completed using the same conditions to provide sufficient material for the composite preparation. An XRD and SEM of the reacted powder were obtained.

9. The samples of SiC coated diamond described in paragraphs 7 and 8, above, were used to prepare aluminum (Al) composites by the high pressure squeeze casting process. This process was chosen over the powder metallurgy composite forming process described in Example 3 of the Miyamoto et al. patent, in order to optimize the thermal conductivity properties of the resulting composites based upon past composite forming experience. The samples of the SiO conversion coated diamond and the CVD coated diamond powders were loaded into separate cavities of a graphite die. The cavities were of a size so as to produce a composite sample of ~0.5" diameter with a thickness of ~0.25". The die was loaded into a high pressure squeeze casting press. The processing parameters were a melt temperature of 850°C, die temperature of 625°C, and an infiltration pressure of 4 Ksi. After the casting process was completed, the die was removed from the press, and the excess Al surrounding the casting was removed by saw cutting and milling, with a final wet grinding until the Al infiltrated diamond was fully exposed on both faces of the samples. The loading of coated diamond in the Al was ~44%(volume).

10. The Al/diamond composite samples described in paragraph 9, above, were sent to Prof. Richard Hasselman at Virginia Polytechnic Institute for measurement of thermal diffusivity. Dr. Hasselman is a recognized expert in the theory and measurement of thermal conductivity of materials. Thermal diffusivity values were converted to thermal conductivity levels using the relationship $K_c=\rho.\kappa_c.h$, where ρ is density of composite, κ is thermal diffusivity h is specific heat, and K is thermal conductivity. Average density of the composite, ρ_c, was estimated using Archimedes principle and corresponded to

3.1g/cm3. Specific heat of the specimens, h_c, was directly measured by calorimetry and corresponded to 710J/k.kg. Measured values of thermal diffusivity and calculated values of thermal conductivity are shown below:

Sample	κ_c	K_c
Conversion coating	0.64 cm^2/sec	141 W/mk
CVD coating	0.65 cm^2/sec	143 W/mk

11. The above results show that the metal matrix composite materials containing SiC coated diamond particles as described in the Miyamoto et al. U.S. Patent No. 6,673,439, do not necessarily or inherently exhibit thermal conductivity characteristics approaching the levels specified in the claims of the above-identified U.S. Patent Application, i.e., greater than about 300 W/mk to greater than about 600 W/mk. This is so whether the SiC coated diamond particles are prepared using the Miyamoto et al. process for conversion coating of diamond with SiO or using the CVD coating process described by Miyamoto et al. for comparison purposes, and even when optimizing the composite forming process for achieving maximal thermal conductivity characteristics.

I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true, and further that these statements are made with the knowledge that willful false statements and the like so made are punishable by fine, or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Roger S. Storm

8/9/05

Date

Signed in Tucson, Arizona